Heterostructures in GaInP grown using a change in Te doping

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In organometallic vapor phase epitaxy, changes in growth conditions can be used to modulate the extent of CuPt ordering and, hence, the band gap energy of GaInP. One method is to add Te during growth. An increase in the band gap energy of 0.1 eV due to a decrease in ordering has been obtained by increasing the input pressure of diethyltelluride from 0 to 8×10^{-6} Torr, which corresponds to a doping concentration of $6\times10^{17}\,cm^{-3}$. This simple procedure offers an attractive method to grow quantum wells (QWs) and superlattices, which are useful for band gap engineering, by modulating the input pressure of the Te precursor. Various heterostructures with abrupt interfaces were successfully grown with interruptions at the interfaces between the Te-doped and undoped GaInP layers. QWs as thin as 10 nm can be clearly seen from transmission electron microscope images. © *2000 American Institute of Physics.* [S0021-8979(00)07411-9]

I. INTRODUCTION

CuPt ordering, the spontaneous segregation of atoms in a ternary alloy into alternating {111} planes, is a phenomenon observed in many semiconductors including essentially all III/V alloys.[1] It is of practical interest because CuPt ordering has a significant effect on the band gap energy. For example, the band gap energy of $Ga_{0.52}In_{0.48}P$ is found to be 160 meV lower in partially ordered materials than in totally disordered materials.[2] Thus, ordering is normally avoided in visible light emitting devices in order to produce the highest emission energies.[3] However, in InAsSb alloys, the shrinkage of band gap energy associated with CuPt ordering is potentially beneficial, since it moves the wavelength further into the infrared where an atmospheric window exists between 8 and 12 μm.[4] Thus, ordered InAsSb has the potential to be a useful material for infrared detectors, if ordering could be precisely controlled. On the other hand, ordering also offers the possibility of producing heterostructures by changing the band gap energy without altering the solid composition. Such structures might be useful for devices such as lasers, light emitting diodes[5] and high efficiency solar cells.[6]

Ordering is a topic of current research interest not only because of the significant technological implications, but also because of the fascinating materials issues, including the thermodynamic driving force and the formation mechanisms. In the bulk, the CuPt structure is not found to be thermodynamically stable relative to the disordered alloy.[7] Thus, it is generally believed that CuPt ordering is induced by processes occurring at the surface during growth. The thermodynamic driving force for CuPt ordering in GaInP is due to formation of rows of $[\bar{1}10]$ oriented P dimers on the (2×4) reconstructed (001) surface.[8] This has been demonstrated by the recent experimental observation, using surface photo absorption (SPA). The concentration of $[\bar{1}10]$ P dimers on the (001) surface correlates closely with the degree of order produced during organometallic vapor phase epitaxial (OMVPE) growth.[9–12]

It has been suggested that the step structure on the surface during growth may also play a role in the ordering process.[13–15] Misorientation of the GaAs substrate toward the [110] direction to produce $[\bar{1}10]$-oriented steps results in a decrease in the degree of order measured in the resulting GaInP layers.[16] SPA studies indicate that this is mainly due to a decrease in the concentration of $[\bar{1}10]$ P dimers on the surface.[15] The presence of [110]-oriented steps, produced by misorienting the substrate by a few degrees toward the $[\bar{1}10]$ direction, is found to increase the degree of order observed in the resulting GaInP layers grown by OMVPE.[13] It also favors the formation of a B variant of the CuPt structure.[14] This is strong evidence that the steps, themselves, affect the ordering process during OMVPE step-flow growth, where every adatom is incorporated into the crystal at a step edge.

Heterostructures[17,18] and quantum wells (QWs)[19] have been produced using the change in order parameter induced by a change in the growth temperature, with no change in the solid composition. The changes in degree of order and band gap energy were found to be abrupt when the growth temperature was altered. However, as a practical process, using a change in temperature to produce the desired change in order parameter is inconvenient. At each interface, the growth must be interrupted for several minutes to allow the temperature to be adjusted and stabilized. In addition, when one of the layers is grown at a high temperature, such as 720 °C, the group III flow rates must typically be adjusted during the interruption to compensate for a decrease in the In incorporation. The degree of order is also found to be a function of the partial pressure of the P precursor (P_P) during growth.[20] Thus, a much more attractive approach would be to change

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P_P to produce the desired change in the order parameter, although this approach changes the band gap energy significantly less than for changes in temperature. Both heterostructures[21] and QWs[22] grown at 670 °C by modulating P_P showed abrupt changes in order parameter as observed in transmission electron microscopy (TEM) images. Low temperature photoluminescence (PL) spectra show distinct peaks for the ordered and disordered layers.

Te doping has been found to affect the step structure and CuPt ordering in GaInP grown on singular and vicinal (001) substrates by OMVPE.[23–25] For growth at 670 °C, the spacing between the $[\bar{1}10]$-oriented steps, observed by atomic force microscopy (AFM), increased by an order of magnitude as the Te doping concentration was increased from 10^{17} to $10^{18}\,cm^{-3}$. The step spacing between the [110]-oriented steps also increased slightly. The band gap energy, determined from the PL peak energy, increased by 110 meV as the doping concentration was increased over the same range. This indicates that disordering may be controlled by the increased velocity of the $[\bar{1}10]$-oriented steps caused by the addition of Te. The dependence of band gap energy on the Te doping concentration provides another way to produce heterostructures and QWs from ordered and disordered GaInP layers.

The purpose of this article is to present results on the production of abrupt heterostructures from ordered and disordered GaInP by modulating the input pressure of the Te precursor. The first attempts to produce disorder-on-order[26] or order-on-disorder heterostructures by switching the Te precursor in or out of the growth chamber were not successful. TEM images and PL spectra indicate a gradual change in order parameter in the upper layer. Subsequently, an interruption in the growth was applied at the interface, before growing the upper layer. TEM images show that the interruptions produced abrupt changes in the order parameter at the heterostructure. The PL spectra also indicate two distinct layers in the heterostructure with order parameters similar to those of single layers. Results from heterostructures made with and without interruption will be presented in order to emphasize the importance of interruptions for producing abrupt interfaces. Double heterostructures (DHs) and QWs were also grown with interruptions to test the usefulness of this technique. Such structures are potentially useful in band gap engineering to produce advanced devices.

II. EXPERIMENTAL PROCEDURE

The GaInP epitaxial layers were grown in a horizontal, atmospheric pressure OMVPE system.[27] Trimethylgallium (TMGa) and trimethylindium (TMIn) were used as the group III precursors with constant bath temperatures of 7.0 and 25.0 °C, respectively. Tertiarybutylphosphine (TBP) was used as the P precursor and kept in the same temperature bath as the TMGa. Diethyltelluride (DETe), diluted to 5 ppm with H_2, was used as the dopant precursor. The substrates used were semi-insulating (001) GaAs misoriented by 3° towards the (111)B direction. Before growth, the substrates were degreased in trichloroethylene, acetone, methanol, and de-ionized (DI) water. The substrates were etched for 1 min in a solution of $12H_2O{:}2NH_4OH{:}1H_2O_2$ followed by a DI water rinse for 5 min and then blown dry with N_2, before being loaded into the system. The growth temperature was 670 °C and the total flow rate was 4 standard liters per minute. The input pressure of TBP was 3 Torr and the V/III ratio was 150. The growth rate was ~0.6 μm/h. For Te-doped GaInP, the input partial pressure of DETe was 6×10^{-6} Torr, which corresponds to a doping concentration of $4\times10^{17}\,cm^{-3}$.[24] Before growing GaInP, a 0.05-μm-thick GaAs buffer layer was deposited to improve the quality of the epilayer. All of the layers discussed here had mirror-like surfaces. No cracks or crosshatching were observed using Nomarski phase contrast optical microscopy. X-ray diffraction was used to measure the lattice constant of the GaInP along the direction perpendicular to the substrate. Assuming that the misfit strain of the GaInP layer was coherently accommodated, the lattice constant of the strain-free layer could be determined.[28] Further, using Vegard's law, the solid composition of GaInP layer was obtained.

For the heterostructures grown without interruption, the procedure used at the interfaces between the ordered and disordered layers was simply to switch DETe in or out of the growth chamber, respectively. On the other hand, for the heterostructures grown *with* interruptions, the TMGa and TMIn were switched out of the growth chamber for 10 min. For the disorder-on-order and order-on-disorder heterostructures DETe was switched in or out, respectively, of the growth chamber at the beginning of the interruption. The flow rates of the group III and group V precursors were the same for both the ordered and disordered layers in the heterostructures grown with or without interruptions.

[110] cross-sectional TEM samples were prepared by standard Ar ion milling at 77 K. The thickness of the sample after ion milling was in the range of 150–400 nm. Dark-field (DF) images taken from the $1/2(\bar{3}3\bar{1})$ superspot were obtained with a JEM 2010 TEM operated at 200 kV. Low temperature PL spectra were measured at 20 K with an excitation intensity of 40 W/cm^2. The 488 nm light from an Ar ion laser was used to excite the samples. Standard lock-in amplifier techniques were used to measure the PL signal.

III. RESULTS

In order to avoid crosshatching and cracks in an epilayer that is lattice mismatched to a substrate, the thickness of the epilayer must be less than the critical thickness.[29,30] Usually for an epilayer with a thickness over 0.2 μm, the lattice mismatch, $f=(a_{sub}-a_{epi})/a_{epi}$, is required to be less than 0.1%. Table I lists the values of solid composition and lattice mismatch for the individual layers of order-on-disorder heterostructures grown with and without interruption. These results indicate that to produce lattice matched heterostructures from ordered and disordered GaInP layers, the group III flow rates must be adjusted during growth to compensate for the change in solid composition with the addition of Te. The change in solid composition from the ordered to the disordered layers is more dramatic in the heterostructures grown with an interruption than in those grown without interruption.

TABLE I. Summary of the growth rate (r_g), the ratio of input pressures of Ga and In precursors [$(Ga/In)^v$], the Ga solid composition (X_{Ga}), and the lattice mismatch between the epilayer and the substrate [$f=(a_{sub}-a_{epi}/a_{epi})$] for heterostructures produced by modulating the DETe flow rate during growth.

Order-on-disorder heterostructure		r_g (μm/h)	$(Ga/In)^v$	X_{Ga}	f (%)
Without interruption	Ordered layer	0.6	0.66	0.51	−0.04
	Disordered layer	0.6	0.66	0.53	0.13
With interruption	Order layer	0.6	0.66	0.50	−0.15
	Disordered layer	0.6	0.66	0.54	0.15

Figure 1(a) shows a DF image from a superspot of a disorder-on-order heterostructure grown without interruption. In this image, the interface cannot be distinguished. However, since both the disordered and ordered layers were grown with the same thickness, the position of the interface was estimated to be in the center of the GaInP layers. The DF image of an order-on-disorder heterostructure grown without interruption is shown in Fig. 1(b). The position of the interface was also estimated, since the interface cannot be seen in the image. Generally, ordered layers appear brighter in a DF image made from a superspot. The upper layer shown in Fig. 1(b) appears to have a degree of order similar to that of the Te-doped lower layer. This implies that the addition of Te gradually changes the degree of order of a GaInP layer.

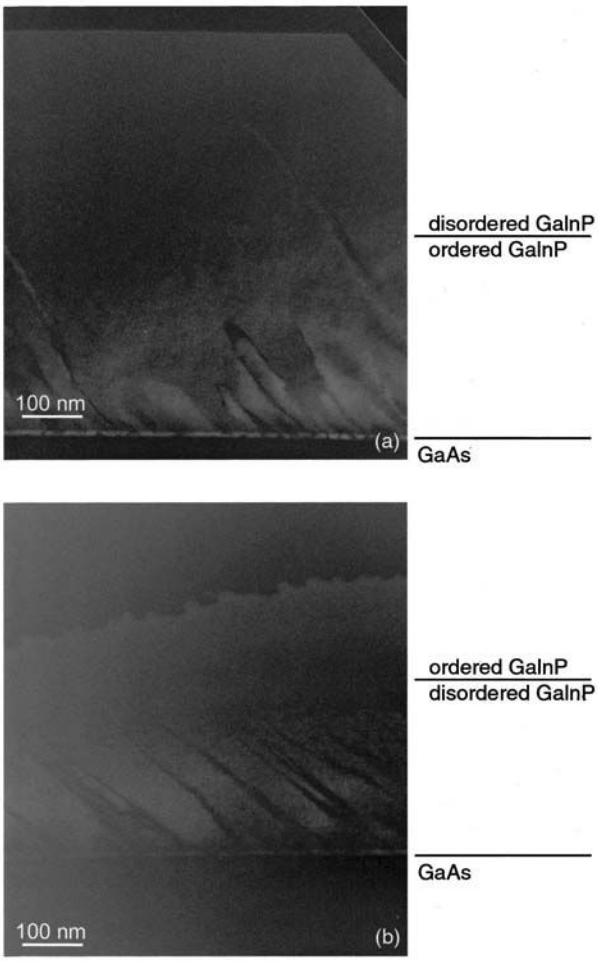

FIG. 1. [110] pole TEM DF images from the 1/2($\bar{3}3\bar{1}$) superspot for disorder-on-order (a) and order-on-disorder (b) heterostructures. Both heterostructures were grown at 670 °C on semi-insulating GaAs, misoriented by 3° from (001) towards the [111]B direction, *without* interruption at the interface between the disordered and ordered layers. The expected positions of the interfaces are indicated.

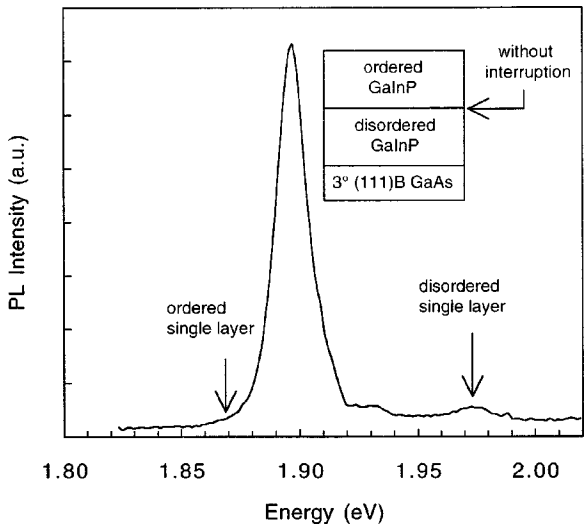

FIG. 2. 20 K PL spectrum and schematic diagram from an order-on-disorder heterostructure grown without interruption. The arrows indicate the peak energies observed for disordered and ordered single layers grown using the same conditions as for the heterostructure.

Figure 2 shows the PL spectrum and a schematic diagram for the order-on-disorder heterostructure shown in Fig. 1(b). The PL is dominated by a single broad peak having an energy between those measured for ordered and disordered single layers grown using the same conditions. This indicates a gradual decrease in the degree of order when Te is added and a gradual increase in the degree of order when Te is removed. The PL results for the disorder-on-order heterostructure were similar to the results for the order-on-disorder heterostructure shown in Fig. 2.

Figures 3(a) and 3(b) show the DF images from superspots for disorder-on-order and order-on-disorder heterostructures grown with a 10 min interruption at the interface. It is clearly seen that the ordered layers are brighter than the disordered layers in both DF images. These results are consistent with the results obtained from single layers that indicate the degree of order decreases with an increase in the Te doping concentration.[23–25] In the heterostructures grown with interruption (Fig. 3), the interfaces between the ordered and disordered layers are abrupt and clearly defined. This is in distinct contrast to the graded interfaces seen in the heterostructures grown without interruption (Fig. 1). This indicates the importance of interruptions in producing abrupt heterostructures. The dark, slanted, lines that thread through the GaInP layers are antiphase boundaries (APBs). These occur when the {111} Ga and In planes in ordered (or partially ordered) material shift out of phase so that the Ga planes line up with the In planes. The fact that APBs can be seen in the Te-doped GaInP layers indicates that this material is still

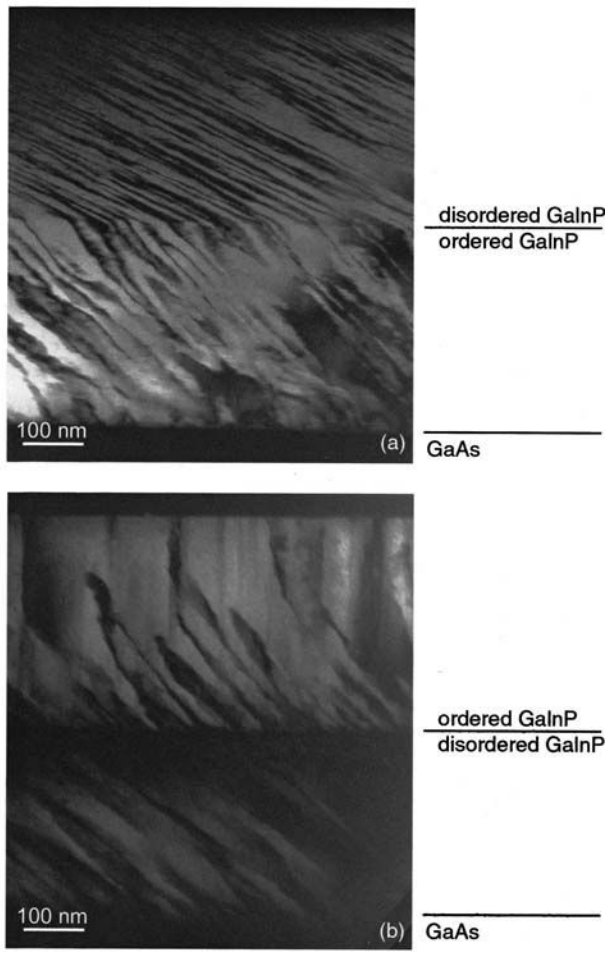

FIG. 3. [110] pole TEM DF images from the $1/2(\bar{3}3\bar{1})$ superspot. Both heterostructures were grown at 670 °C on semi-insulating GaAs, misoriented by 3° from (001) towards the [111]B direction, *with* a 10 min interruption at the interface between the disordered and ordered layers. The interfaces in the disorder-on-order heterostructure (a) and in the order-on-disorder heterostructure (b) can be clearly seen. The dark, slanted lines that appear to thread through the heterostructures are antiphase boundaries.

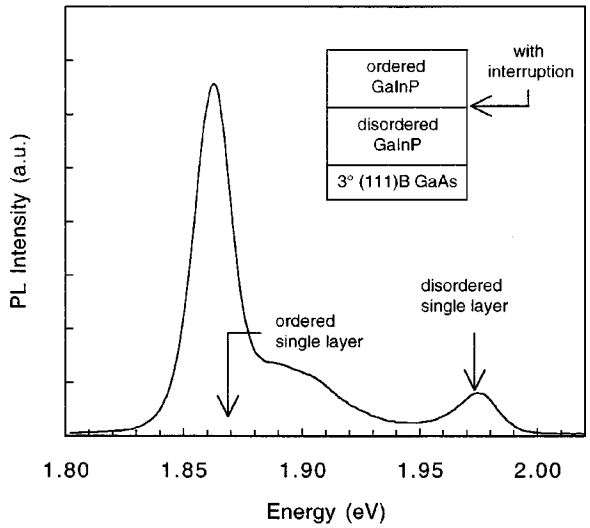

FIG. 4. 20 K PL spectrum and schematic diagram from an order-on-disorder heterostructure grown with a 10 min interruption between the layers. The arrows indicate the peak energies observed for disordered and ordered single layers grown using the same conditions as the heterostructure.

partially ordered, although it is much less ordered than the undoped GaInP.

Figure 4 shows the PL spectrum and a schematic diagram for the order-on-disorder heterostructure grown with an interruption shown in Fig. 3(b). Two PL peaks are observed at the energies, indicated by the arrows in the figure, measured for the ordered and disordered single layers. Figure 4 clearly shows that two distinct layers were produced in the heterostructure, similar in degree of order to ordered and disordered single layers. The shoulder on the low energy PL peak is most likely due to a partially ordered layer next to the GaAs substrate. The fact that the peak is broad suggests a varying degree of order. As can be seen in most of the DF images of the heterostructures, the first layer, next to the substrate, is often partially ordered. The PL result for the disorder-on-order heterostructure grown with an interruption confirms the results for the order-on-disorder heterostructure shown in Fig. 4. The DF images (e.g., Fig. 3) and the PL spectra (e.g., Fig. 4) clearly show that abrupt order-on-disorder and disorder-on-order heterostructures can be produced with the addition of 10 min interruptions at the interfaces during growth.

Several disorder/order/disorder DHs and QWs were grown using the same interruption technique used to produce the abrupt single heterostructures. During the interruption, the TMIn flow rate was adjusted slightly to compensate for the change in solid composition between the ordered and disordered layers. Consequently, both layers were lattice matched to the substrate. Figures 5(a) and 5(b) show the [110] TEM DF cross-sectional images for a nominal 32-nm-thick DH and a nominal 10-nm-thick QW, respectively. The ordered wells in these images are clearly seen to be brighter than the disordered barriers, consistent with the results shown in Fig. 3. Careful examination of Figs. 5(a) and 5(b) indicates variations in the thickness of the thin, ordered layers. The fact that the bright, thin, ordered layers are sandwiched between darker, disordered layers makes the thickness variation clearly observable. Variations at the interfaces of single heterostructures are not obvious in the DF images, but are likely present. In Fig. 5(b), the thickness variation appears to be due to facets at the interface.

Figures 6(a) and 6(b) show the 20 K PL spectra for the DH and the QW, respectively. The inset shows a schematic diagram of the disorder/order/disorder heterostructures. In Fig. 6(a), a small PL peak is observed near the energy measured for the disordered single layer indicated by the arrow. This suggests that the Te-doped layers in the DH are not as disordered as the Te-doped single layer. A dominant PL peak appears at a lower energy of ~1.91 eV. This peak is most likely from the thin ordered layer, although it clearly indicates that this layer is not as ordered as an undoped single layer. The two PL peaks, clearly show that distinct ordered and disordered layers can be produced in a DH grown with interruptions.

Figure 6(b) shows the PL spectrum from the 10-nm-thick QW shown in Fig. 5(b). The growth conditions for the

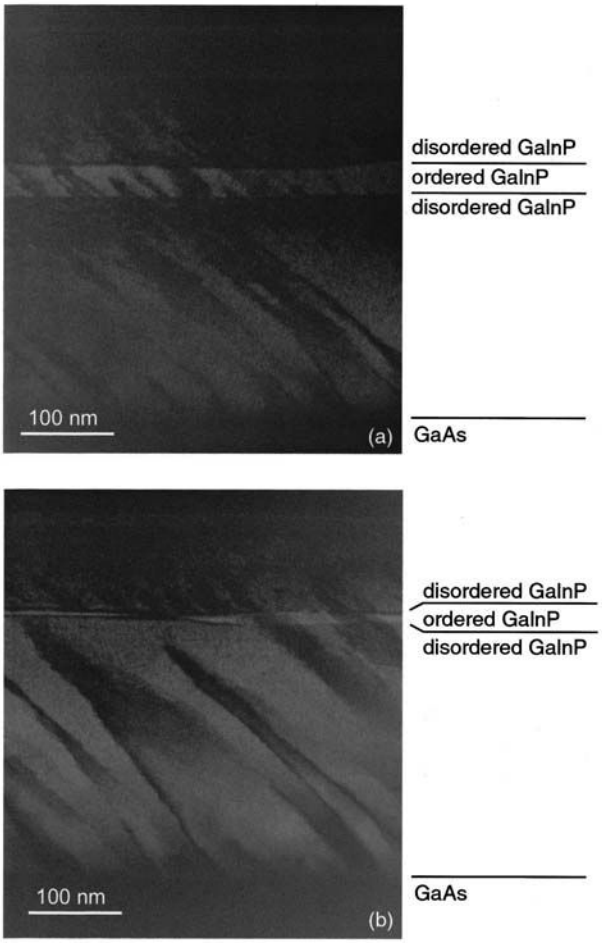

FIG. 5. [110] pole TEM DF images from the $1/2(\bar{3}3\bar{1})$ superspot obtained from a disorder/order/disorder DH (a) and a QW (b) grown on semi-insulating GaAs, misoriented by 3° from (001) towards the [111]B direction, *with* 10 min interruptions at both interfaces. The nominal thickness of the ordered layer in the DH is 32 nm and in the QW is 10 nm.

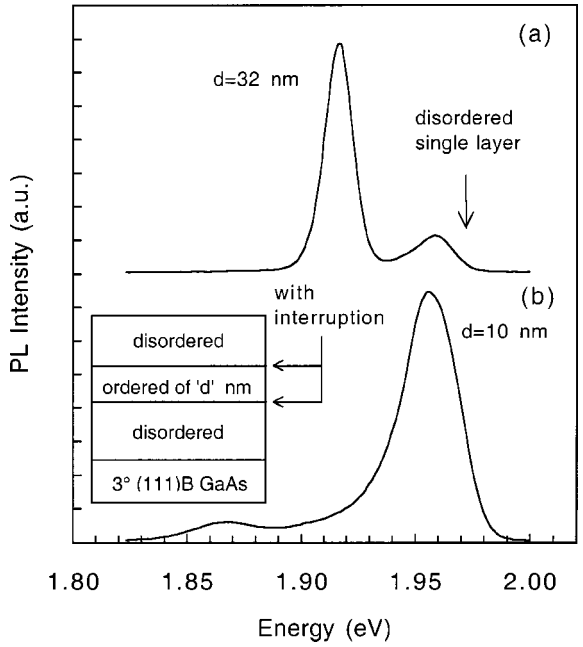

FIG. 6. 20 K PL spectra and schematic diagram from the disorder/order/disorder DH (a) and QW (b). The nominal well thickness is 32 nm in the DH and 10 nm in the QW. The arrow indicates the peak energy observed for a disordered single layer grown using the same conditions.

QW were the same as those for the DH. The spectrum in Fig. 6(b), including the small peak at 1.86 eV, corresponds closely to the spectrum for a disordered single layer. Thus, in contrast to Fig. 6(a) that shows a PL peak from the well, Fig. 6(b) shows no PL peak appearing at the energy expected for a 10-nm-thick well. A possible explanation for this will be discussed in the next section.

IV. DISCUSSION

Te doping significantly affects the step structure and CuPt ordering in GaInP on singular and vicinal (001) substrates.[23–25] A qualitative model[25] has been proposed to explain this behavior, based on the assumption that Te collects at the step edges. The extra valence electron of Te, relative to the P that it replaces, certainly changes the results of electron counting at the surface and thus, at high concentrations, removes the driving force for formation of the (2 $\times$2) reconstruction at the {111}A and {111}B step edges.[24] This is postulated to result in the formation of mainly monolayer steps on singular surfaces. Presumably, the dangling bonds due to Te atoms on the bunched steps destabilize these steps and stabilize the monolayer steps. This has been observed experimentally by AFM.[24] The monolayer steps formed at high Te doping concentrations could account for very high group III sticking coefficients, due to the possible formation of three bonds for a group III adatom at the step edge.[24] An increase in the sticking coefficient with the addition of Te could account for the 20$\times$ increase in the $[\bar{1}10]$ step spacing[26] and also the decrease in the degree of order of the resulting layer. This same model can also be used to explain the data for the single heterostructures grown with and without interruption.

Both the TEM image [e.g., Fig. 1(b)] and the PL spectrum (e.g., Fig. 2) from the order-on-disorder heterostructure indicate that the upper layer had a gradual increase in the degree of order, resulting in a graded heterostructure. A possible explanation for the gradual increase in the degree of order is that the Te concentration on the surface decreases gradually during growth. Thus, the surface step structure of the lower layer grown with a high Te doping concentration would persist into the upper layer. This gradual change could result in a gradual increase in the ordering of the upper layer. On the other hand, when an interruption is applied before growing the upper layer, the Te atoms that have collected at the step edges during the growth of the lower layer will desorb and the step structure will consequently change. After the interruption, a more uniform ordered layer, similar to the ordered single layer, is produced. This is confirmed by the TEM images [e.g., Figs. 3(a) and 3(b)] that show abrupt interfaces and by the PL results (e.g., Fig. 4) that show the upper layer has the same degree of order as an ordered single layer grown with the same conditions.

The model can also be used to explain the difference between the disorder-on-order heterostructures grown with

and without interruption. When the disordered layer is continuously grown on the ordered layer, the Te concentration on the surface builds up gradually. The result is that the bunched steps formed during the growth of the lower layer persist into the upper layer. Thus, during the growth of the upper layer, a gradual decrease in ordering is expected. The TEM DF images [e.g., Fig. 1(a) and 1(b)] clearly show the gradual change in ordering. However, with a 10 min interruption, most of the change in Te concentration and surface structure occurs before growth of the second layer. Indeed, a much more abrupt disorder-on-order heterostructure results, as observed in the DF images [e.g., Figs. 3(a) and 3(b)].

For a 10-nm-thick QW, the PL spectrum [e.g., Fig. 6(b)] shows a broad PL peak near the energy measured for the disordered single layer. The difference in peak energy for the DH compared to the single layer is most likely due to a small change in the solid composition of the DH that was implemented to improve the lattice matching of the layers to the GaAs substrate. Broadening of the dominant PL peak suggests that there is significant variation in the degree of ordering in the layers. No luminescence from the thin well is visible. This may be due to nonuniformity in the well thickness, as seen in Fig. 5(b). Varying thickness will broaden the PL from the well and decrease the intensity accordingly. This suggests that the PL emission from the well may be buried under the low energy tail of the dominant PL peak from the disordered layer(s).

V. SUMMARY

Abrupt heterostructures made by changing the order parameter in CuPt-ordered GaInP were demonstrated by modulating the input pressure of the Te precursor between 0 and 6×10^{-6} Torr. The heterostructures were grown by OMVPE at 670 °C on semi-insulating GaAs substrates misoriented by 3° from (001) toward the [111]B direction. In the single heterostructures grown without interruption between the ordered and disordered layers, the interfaces are not clearly defined in the TEM DF images, due to a gradual change in order parameter. In single heterostructures grown with a 10 min interruption, the interfaces are more abrupt as seen in the DF images. PL spectra from the heterostructures grown with interruptions show two distinct PL peaks with energies near those measured for single layers. Both the TEM and PL results indicate that abrupt heterostructures can be produced when interruptions are used. This technique was used to produce disorder/order/disorder DHs and QWs. The PL spectrum from the DH shows two distinct peaks corresponding to those measured for ordered and disordered single layers.

ACKNOWLEDGMENTS

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